Sudan University of Science and Technology
College of sciences
Department of Chemistry

A research Submitted in Fulfillment for the Requirement of Bachelor Degree in English Language

Determination of Lead Concentration in Cosmetic (Kohl)

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الآية

قال تعالى:

﴿ وَقُل رَّبِّ زِدْنِي عِلْمًا ﴾

صدق الله العظيم
سورة طه، الآية (114)

Dedication

This project is dedicated to our parents for their countless sleepless nights filled with prayers for our success in our life , the least we can do is to dedicate the fruit of our efforts for the most two influential people in our life, this is for you.

Our professors and teachers who have given us all their renewed support with singularly honest, Thank you for bring us to this stage.

our colleagues who gave us those wonderful moments in the arena of scientific, socialand cultural.

To all those with whom we have shared life difficulties, we will never really find those perfect words to thank you.

Acknowledgment

First and foremost I would like to thanks Allah for all the blessing he have bestowed upon me , and for keeping me strong when time were hard and for giving me the wisdom which has helped me to become the person I am today.

I'd like express my deep thanks to my supervisor Dr. Elmugdad Ahmed Ali.

To whom I'm overwhelmed with gratitude for this efforts and supports.

Abstract

Was made studying to measure the concentration of Lead element in cosmetics (Khol). Which was taken different samples and was made the primary conducted processors then was measured the concentration of Lead element and the Absorptivity by atomic absorpation which was included analysis of different types of Kohl (powder- gel –pencil).

Analysis Laboratories have been the Department of Chemistry – Faculty of Science- Sudan University of Science and Technology and Central Laboratory- Faculty of Science in Khartoum University.

مستخلص البحث

اجريت هذه الدراسه لقياس تركيز عنصر الرصاص في مستحضر التجميل (الكحل) .اخذت عينات مختلفه من الكحل و تم اجراء المعالجات الاولية و من ثم قياس تركيز عنصر الرصاص و قياس الامتصاصية عن طريق جهاز الامتصاص الذري و يتضمن تحليل عينات من انواع مختلفه من الكحل (البدرة –الجل و القلم) تم التحليل بجامعه السودان للعلوم و التكنولوجيا و المعمل المركزي بجامعه الخرطوم .

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Chapter One

1.1 Introduction Literature Review:

Kohl is a cosmetic predominantly used as an eye makeup it is widely used by woman ,man and on children and babies in Asia , the middle east , and most areas of Africaexcept south Africa . it is worn for a variety of reasons including tradition beautification , to ward off the "evil eye " the widespreaisd belief that kohl is medically beneficial for the eyes and finally because wearing kohl is encouraged within the sunna , the traditional behavioral guidelines of the Islamic religion.

This study was undertaken to ascertain whether this substance is a possible source of lead exposure , to determine if individuals from third – world nations were continuing to use this product in first world nation and to confirm commercial availability of this substance in first – world nations.

1.2 Kohl:

Is an ancient eye cosmetic, traditionally made by grinding stibnite (Sb_2S_3). It is widely used in South Asia, the Middle East, North Africa, the Horn of Africa, and parts of West Africa as eyeliner to contour and/or darken the eyelids and as **mascara** for the eyelashes. It is worn mostly by women, but also by some men and children. Marketed as "kohl", but are prepared differently and in accordance with relevant health standards.

Kohl (from the ancient Semitic root k-H-l; Biblical Hebrew kajl, Arabic kohl)

"Blue" Kohl is a dark-bluish black pigment composed of both lead-based compounds as well as a compound of antimony. The lead-based compounds in kohl are galena (PbS) – dark grey and gloss laurionite(PbCl(OH)) – white phosgenite ($(PbCl)_2CO_3$); cerussite ($PbCO_3$) – blue. The antimony-based compound in kohl is stibnite (Sb_2S_3) - blue. Stibnite has antimony (stibium) in it.

There is evidence that submicromolar concentrations of lead (Pb^{2+}) can elicit overproduction of nitrous oxide (N_2O). Kohl was used not only as a cosmetic but also as a medicinal collyrium (from Gr. kollurion). Two of kohl's lead compounds - the lead chlorides laurionite and phosgenite - were not natural to the Nile valley. It is believed they were intentionally synthesized by the ancient Egyptians for this purpose. The widespread use of kohl across the Mediterranean and the Mid-east attests to its ability to protect the eye from infectious disease and be used as a cosmetic.

1.2.1 Kohl composition:

1.2.1.1 Legal status:

Kohl is illegal to import into or sell in the United States, because it is not on the list of color additives approved by the Food and Drug Administration, which considers kohl unsafe for use.

1.2.1.2 GALENA:

Galena also called lead glance, is the natural mineral of lade (II) sulfide. It is most important ore of lead and an important source of silver.

Galena is one of the most abundant distributed sulfide minerals .it crystallizes in the Cubic crystal system, octahedral sulfide group of minerals metal ions in octahedral positions.

1.2.1.2. A Galena uses:

One of the oldest uses of galena was as Kohl, which, in ancient Egypt, was applied around the eye to reduce the glare of the desert sun and to repel flies, which were potential source of disease.

Galena is primary ore of lead, which is mainly used in making lead-acid batteries, lead sheets and shots.

1.2.1.2. B Health issues:

Galena contains lead, a toxic element. While bound to crystal structure, the lead content of galena is of minor concern and the mineral is safe to handle. However, prolonged exposure via inhalation or ingestion pulverized dust is hazardous to one's health.

1.2.1.3 Lead:

Lead is a chemical element in the carbon group with symbol Pb (from Latin: *plumbum*) and atomic number 82. It is a soft, malleable and heavypost-transition metal. Freshly cut, solid lead has a bluish-white color that soon tarnishes to a dull grayish color when exposed to air; the liquid metal has shiny chrome-silver luster. Lead has the highest atomic number[a] of any non-radioactive element, although the next higher element, bismuth, has one isotope with a half-life that is long enough (over one billion times the estimated age of the universe) to be considered stable for practically all purposes. Lead's four stable isotopes each have 82 protons, a magic number in the nuclear shell model of atomic nuclei. The isotope lead-208 also has 126 neutrons, another magic number, and is hence double magic, a property that grants it enhanced stability: lead-208 is the heaviest known stable nuclide.

Lead is used in building construction, lead-acid batteries, bullets and shot, weights, as part of solders, pewters, fusible alloys, and as a radiation shield.

If ingested or inhaled, lead and its compounds are poisonous to animals and humans. Lead is a neurotoxin that accumulates both in soft tissues and the bones, damaging the nervous system and causing brain disorders. Excessive lead also causes blood disorders in mammals. Lead poisoning has been documented since ancient Rome, ancient Greece, and ancient China.

Fig (1)

General properties

Name, symbol	lead, Pb
Pronunciation	/lɛd/ ***LED***
Appearance	metallic gray
Atomic number(*Z*)	82
Group, block	group 14 (carbon group), p-block
Period	period 6
Element category	post-transition metal

Standard atomic weight (±)(A_r)	207.2(1)[1]
Electron configuration	[Xe] $4f^{14}$ $5d^{10}$ $6s^2$ $6p^2$
per shell	2, 8, 18, 32, 18, 4

Physical properties

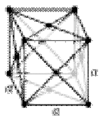
Phase	solid
Melting point	600.61 K (327.46 °C, 621.43 °F)
Boiling point	2022 K (1749 °C, 3180 °F)
Density near r.t.	11.34 g/cm^3
when liquid, at m.p.	10.66 g/cm^3
Heat of fusion	4.77 kJ/mol
Heat of vaporization	179.5 kJ/mol
Molar heat capacity	26.650 J/(mol·K)

vapor pressure

P (Pa)	1	10	100	1 k	10 k	100 k
at T (K)	978	1088	1229	1412	1660	2027

Atomic properties:

Oxidation states	**4**, 3, **2**, 1, −1, −2, −4 (an amphoteric oxide)

Electronegativity	Pauling scale: 1.87
Ionization energies	1st: 715.6 kJ/mol 2nd: 1450.5 kJ/mol 3rd: 3081.5 kJ/mol
Atomic radius	empirical: 175 pm
Covalent radius	146±5 pm
Van der Waals radius	202 pm
Miscellanea	
Crystal structure	face-centered cubic (fcc) 
Speed of sound thin rod	1190 m/s (at r.t.) (annealed)
Thermal expansion	28.9 µm/(m·K) (at 25 °C)
Thermal conductivity	35.3 W/(m·K)
Electrical resistivity	208 nΩ·m (at 20 °C)
Magnetic ordering	diamagnetic
Young's modulus	16 GPa
Shear modulus	5.6 GPa
Bulk modulus	46 GPa

Poisson ratio	0.44
Mohs hardness	1.5
Brinell hardness	38–50 MPa
CAS Number	7439-92-1

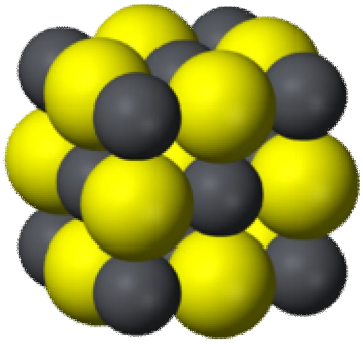
History

Discovery	Middle Easterns(7000 BCE)

Most stable isotopes of lead:

iso	NA	half-life	DM	DE(MeV)	DP
^{202}Pb	syn	53000 y	ε	0.0497	^{202}Tl
^{204}Pb	1.4%	>1.4×10^{20} y[2]	(α)	1.972	^{200}Hg
^{205}Pb	trace	1.53×10^{7} y	ε	0.051	^{205}Tl
^{206}Pb	24.1%	>2.5×10^{21} y[2]	(α)	1.1366	^{202}Hg
^{207}Pb	22.1%	>1.9×10^{21} y[2]	(α)	0.3915	^{203}Hg
^{208}Pb	52.4%	>2.6×10^{21} y[2]	(α)	0.5188	^{204}Hg
^{210}Pb	trace	22.3 y	β^-	0.064	^{210}Bi

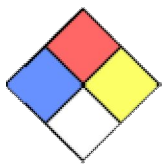
1.2.1.4 Lead (II) sulfide:



Fig(2)

Names:	
Other names	
Plumbous sulfide Galena, Sulphuret of lead	
Identifiers:	
CAS Number	1314-87-0 ✓
ChemSpider	14135 ✗
Jmol 3D model	Interactive image
InChI[show]	
SMILES[show]	

Properties:	
Chemical formula	PbS
Molar mass	239.30 g/mol
Density	7.60 g/cm
Melting point	1,118 °C (2,044 °F; 1,391 K)
Boiling point	1,281 °C (2,338 °F; 1,554 K)
Solubility in water	2.6×10^{-11} kg/kg (calculated, at pH=7) 8.6×10^{-7} kg/kg
Solubility product (K_{sp})	9.04×10^{-29}
Refractive index (n_D)	3.91
Structure:	
Crystal structure	Halite (cubic), cF8
Space group	Fm3m, No. 225
Lattice constant	a = 5.936 Angstroms
Coordination geometry	Octahedral (Pb^{2+}) Octahedral (S^{2-})
Thermo chemistry:	
Specific heat capacity (C)	46.02 J/degree mol

Std molar entropy ($S^{\ominus}_{298}$)	91.3 J/mol
Std enthalpy of formation ($\Delta_f H^{\ominus}_{298}$)	–98.7 kJ/mol
Hazards:	
Safety data sheet	External MSDS
EU classification (DSD)	Repr. Cat. 1/3 Harmful (**Xn**) Dangerous for the environment (**N**)
R-phrases	R61, R20/22, R33, R62, R50/53
S-phrases	S53, S45, S60, S61
NFPA 704	 0 2 0
Flash point	Non-flammable
Related compounds:	
Other anions	Lead(II) oxide

	Lead selenide Lead telluride
Other cations	Carbon monosulfide Silicon monosulfide Germanium(II) sulfide Tin(II) sulfide
Related compounds	Thallium sulfide Lead(IV) sulfide Bismuth sulfide

Lead(II) sulfide (also spelled *sulphide*) is an inorganic compound with the formula PbS. PbS, also known as galena, is the principal ore, and most important compound of lead. It is a semiconducting material with niche uses.

1.2.1.4.1Formation, basic properties, related materials:

Addition of hydrogen sulfide or sulfide salts to a solution of lead ions gives PbS as an insoluble black precipitate.

$$\mathbf{Pb^{2+} + H_2S \rightarrow PbS + 2\ H^+}$$

The equilibrium constant for this reaction is 3×10^6 M. This reaction, which entails a dramatic color change from colourless or white to black, was once used in qualitative inorganic analysis. The presence of hydrogen sulfide or sulfide ions is still routinely tested using "lead acetate paper."

Like the related materials PbSe and PbTe, PbS is a semiconducter. In fact, lead sulfide was one of the earliest materials to be used as a semiconductor. Lead sulfide crystallizes in the sodium chloride motif, unlike many other IV-VI semiconductors.

Since PbS is the main ore of lead, much effort has focused on its conversion. A major process involves smelting of PbS followed by reduction of the resulting oxide. Idealized equations for these two steps are:

$$\mathbf{2\ PbS + 3\ O_2 \rightarrow 2\ PbO + 2\ SO_2}$$

$$\mathbf{PbO + C \rightarrow Pb + CO}$$

The sulfur dioxide is converted to sulfuric acid.Lead sulfide-containing nanoparticle and quantum dots have been well studied. Traditionally, such materials are produced by combining lead salts with a variety of sulfide sources. PbS nanoparticles have been recent examined for use in solar cells.

1.2.1.4.2Applications:

PbS was once used as a black pigment, but current applications exploit its semiconductor properties, which have long been recognized.

1.2.1.4.3 Astronomy:

Elevations above 2.6 km (1.63 mi) on the planet Venus are coated with a shiny substance. Though the composition of this coat is not entirely certain, one theory is that Venus "snows" crystallized lead sulfide much as Earth snows frozen water. If this is the case, it would be the first time the substance was identified on a foreign planet. Other less likely candidates for Venus' "snow" are bismuth sulfide and tellurium.

1.2.1.4.4 Safety:

Lead (II) sulfide is so insoluble that it is almost nontoxic, but pyrolysis of the material, as in smelting, gives dangerous fumes. Lead sulfide is insoluble and a stable compound in the pH of blood and so is probably one of the less toxic forms of lead. A large safety risk occurs in the synthesis of PbS using lead carboxylates, as they are particularly soluble and can cause negative physiological conditions.

1.2.2 Forms of Kohl:

1.2.2.1 Powdered kohl:

In its simplest form, kohl is a fine black or dark grey powder. In the past, better quality kohl was made by grinding up galena (lead sulfide) or stibnite (antimony sulfide) – both of which are poisons – but it could also made from carbon black or iron oxide – which are harmless. Kohl is therefore a chemically diverse material, defined more by its color and use than its composition. In Europe, this diversity was magnified by the tendency to call any black eyebrow or eyelash cosmetic a kohl, even those made from materials such as ink – a situation that lasted up to the Second World War.

The kohl or black eyebrow cosmetic is variously prepared. The spirituous: Lampblack 140 is finely divided in simple tincture of benzoic (20 per cent. strength) 210, and there are added 12.5 of gum lac dissolved in 630 of alcohol, 5 of castor oil, and 2.5 of rose synthetic extra or other perfume as desired. The ordinary Liquid Kohl is made from (a) lampblack, ivory black, or drop black 10, with powdered gum acacia 10, rubbed out thoroughly with 80 of rose water or orange flower water; or (b) the acacia in the last example is replaced by 2 of powdered tragacanth rubbed down with 10 of perfumed spirit. Continental forms of kohl are prepared from Chinese or Indian ink, as (a) Indian ink powder 10, powdered gum acacia 1, and orange flower water to 100; or (b) Indian ink liquid 64, glycerin 15, gum acacia 1 in rose water 5, and perfumed spirit 15. Paste forms of kohl are prepared from about 8 of one of the fine blacks, rubbed out fine with 1 of hard and 11 of soft paraffin previously melted.(Eye Cosmetics, 1932)

1.2.2.1.1 Using powdered kohl:

In the East, powdered kohl is traditionally used as an eyeliner and eyeshadow but it is not always applied in the way that most people apply these products in the West. In the European tradition it is more common to line the outside of the eyelashes – above the upper lashes and below the lower ones. In the East powdered kohl can be smudged around the eye but it is also traditionally applied on inner surface of the eyelid – known as the conjunctiva, inner rim or waterline.

The technique used to apply kohl to the waterline involves dipping a rod-like applicator into kohl powder and then, after flicking off the excess, passing the rod through the base of the lashes while the eyes are closed. If done correctly, the wet conjunctiva picks up the powder from the side of the rod creating a smooth, even line. Any spots that are missed can then be lightly touched up with more powder applied using the pointed end of the applicator

1.2.2.2Paste kohl:

In the East, powdered kohl was also made into a paste by mixing it with fats and oils. The paste was traditionally applied on the eyebrows and around the eyes so was equivalent to Western eyeshadows, eyeliners or mascaras. It is possible that this is what the Ballets Russes dancers and silent screen Vamps used, but it is hard to be sure. Given that stage and screen performers of the time had a wide variety of materials that could be used to blacken the eyes – including greasepaint liners, Indian or Chinese ink mixed into glycerol, lampblack incorporated into a fat or gum, and mascaro/water cosmetique – and the tendency to call any black eyebrow or eyelash cosmetic a kohl, it is difficult to be certain about the cosmetics they employed.

It seems likely that more than one cosmetic was used to achieve the effect they were after and kohl may not have been used at all. Helena Rubinstein – who claims to have been responsible for Theda Bara's eye make-up – makes no mention of it.

For Theda Bara, the Siren of the Silent Screen, we helped to create 'The Vamp' look which became internationally famous. Enthusiastic fans copied her hairstyles, her clothes, and her mannerisms. Her eyes were amazingly beautiful, but camera techniques were not as advanced as they are now and much of her beauty was lost on the screen. She came to me to find a way of emphasizing them.

Eye make-up of any kind was unknown in America. Mascara had been used only in France by a few stage personalities, and not always well. But with my love for the theatre and my insatiable curiosity, I had delved into the beauty secrets of several French actresses. I had also experimented with kohl (the eye make-up invented by the ancient Egyptians and used by Cleopatra). For Theda Bara I made a mascara which drew attention to her lovely eyes so that they dominated her whole face—and the mascara did not streak! I also added a touch of colour to her eyelids. The effect was tremendously dramatic. It was a sensation reported in every newspaper and magazine—only a little less of a sensation than when Theda Bara first silvered her eyelids!

1.3 Atomic absorption spectroscopy Principles:

The technique makes use of absorption spectrometry to assess the concentration of an analyte in a sample. It requires standards with known analyte content to establish the relation between the measured absorbance and the analyte concentration and relies therefore on the Beer-Lambert Law. In short, the electrons of the atoms in the atomizer can be promoted to higher orpitals (excited state) for a short period of time (nanoseconds) by absorbing a defined quantity of energy (radiation of a given wavelength). This amount of energy, i.e., wavelength, is specific to a particular electron transition in a particular element. In general, each wavelength corresponds to only one element, and the width of an absorption line is only of the order of a few picometers (pm), which gives the technique its elemental selectivity. The radiation flux without a sample and with a sample in the atomizer is measured using a detector, and the ratio between the two values (the absorbance) is converted to analyte concentration or mass using the Beer-Lambert Law.

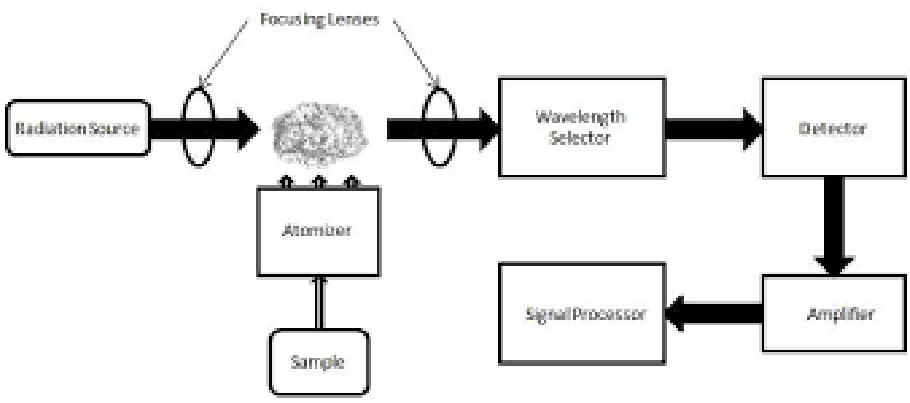


Fig (3)

1.4 Infrared absorption principle:

IR radiation does not have enough energy to induce electronic transitions as seen with UV. Absorption of IR is restricted to compounds with small energy differences in the possible vibrational and rotational states. For a molecule to absorb IR, the vibrations or rotations within a molecule must cause a net change in the dipole moment of the molecule. The alternating electrical field of the radiation (remember that electromagnetic radiation consists of an oscillating electrical field and an oscillating magnetic field, perpendicular to each other) interacts with fluctuations in the dipole moment of the molecule. If the frequency of the radiation matches the vibrational frequency of the molecule then radiation will be absorbed, causing a change in the amplitude of molecular vibration.

1.4.1. Molecular rotations:

Rotational transitions are of little use to the spectroscopist. Rotational levels are quantized, and absorption of IR by gases yields line spectra. However, in liquids or solids, these lines broaden into a continuum due to molecular collisions and other interactions.

1.4.2. Molecular vibrations:

The positions of atoms in a molecules are not fixed; they are subject to a number of different vibrations. Vibrations fall into the two main catagories of *stretching* and *bending*.

1.4.3. Stretching:

Change in inter-atomic distance along bond axis

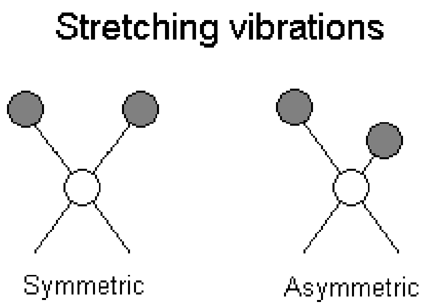


Fig (4)

1.4.4. Bending:

Change in angle between two bonds. There are four types of bend:

- Rocking.
- Scissoring.
- Wagging.
- Twisting.

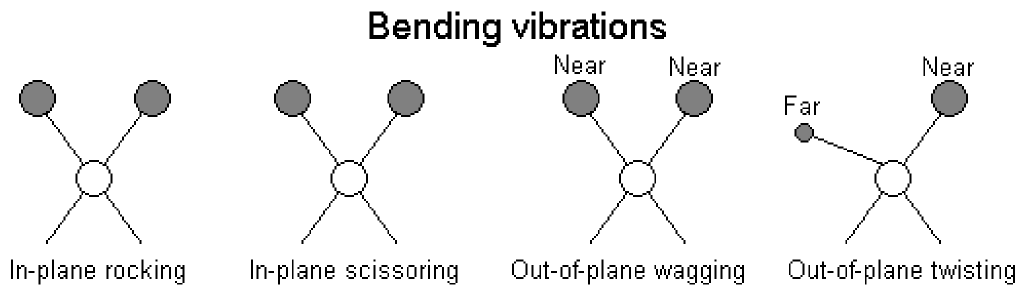


Fig (5)

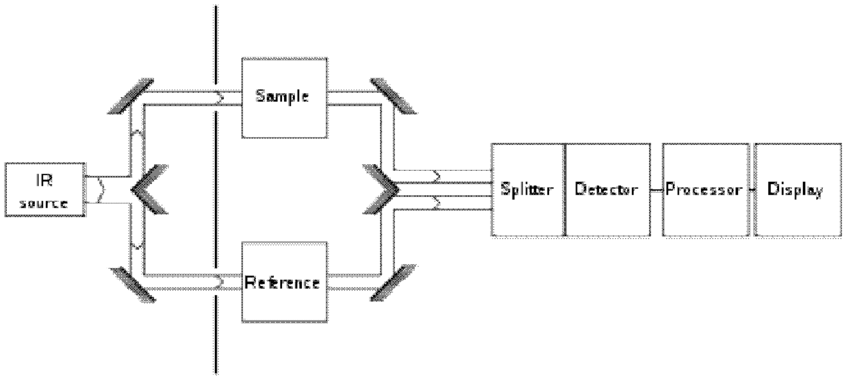

IR
source
Sample
Reference
Splitter
Detector
Processor
Display

Fig (6)

Chapter Two

2.1 Objective:

Determination of concentration of lead as lead sulfide(galena) in kohl.

2. 2 Sampling:

Five samples were taken randomly from market . two as powder (athmade , green kohl) ,and two as pencil (lorial Paris , xxl) last one as gel(kajl).

2.3 Materials:

- Samples.
- Hydrochloric acid (5M).
- Nitric acid.
- Distilled water.

2.4Appliance and tools:

- Atomic absorption (shimadzu AA6800 Atomic absorption spectrophotometer)
- Furnace
- Water bath
- Mortar or pastle
- Silica dishes
- Glass rode
- Dropper
- Beakers (100ml)
- Volumetric flask(50ml)

2.5 Sample pre-treatment:

Weight of samples was finely grounded by mortar and dried in furnace at 450 C for 3 hour, and determine of heavy metal (lead).

2.6 Preparation of solutions:

0.2g of burned sample was weighed in to 100ml beakers , dissolved by 15ml of Hydrochloric solution (5M) ,few drops of Nitric acid was added and evaporated to dryness in water bath , the sample was redissolved in 5M of HCl, warmed , filtered into a50ml volumetric flask and completed to mark with distilled water .

Chapter Three

3.1 Results:

	Sample ID	Pb: Abs	Pb: Actual Conc.%	Pb: Actual Conc. unit
1	Kajl	0.0006	0.0412	Ppm
2	Athmed	0.0415	284.8100	Ppm
3	Nour	0.0022	0.1510	Ppm
4	L'Oreal	0.0050	0.3431	Ppm
5	Xxl Pencil	0.0042	0.2882	Ppm

3.2 Characterization of lead:

3.2.1 Infrared spectroscopy of lead:

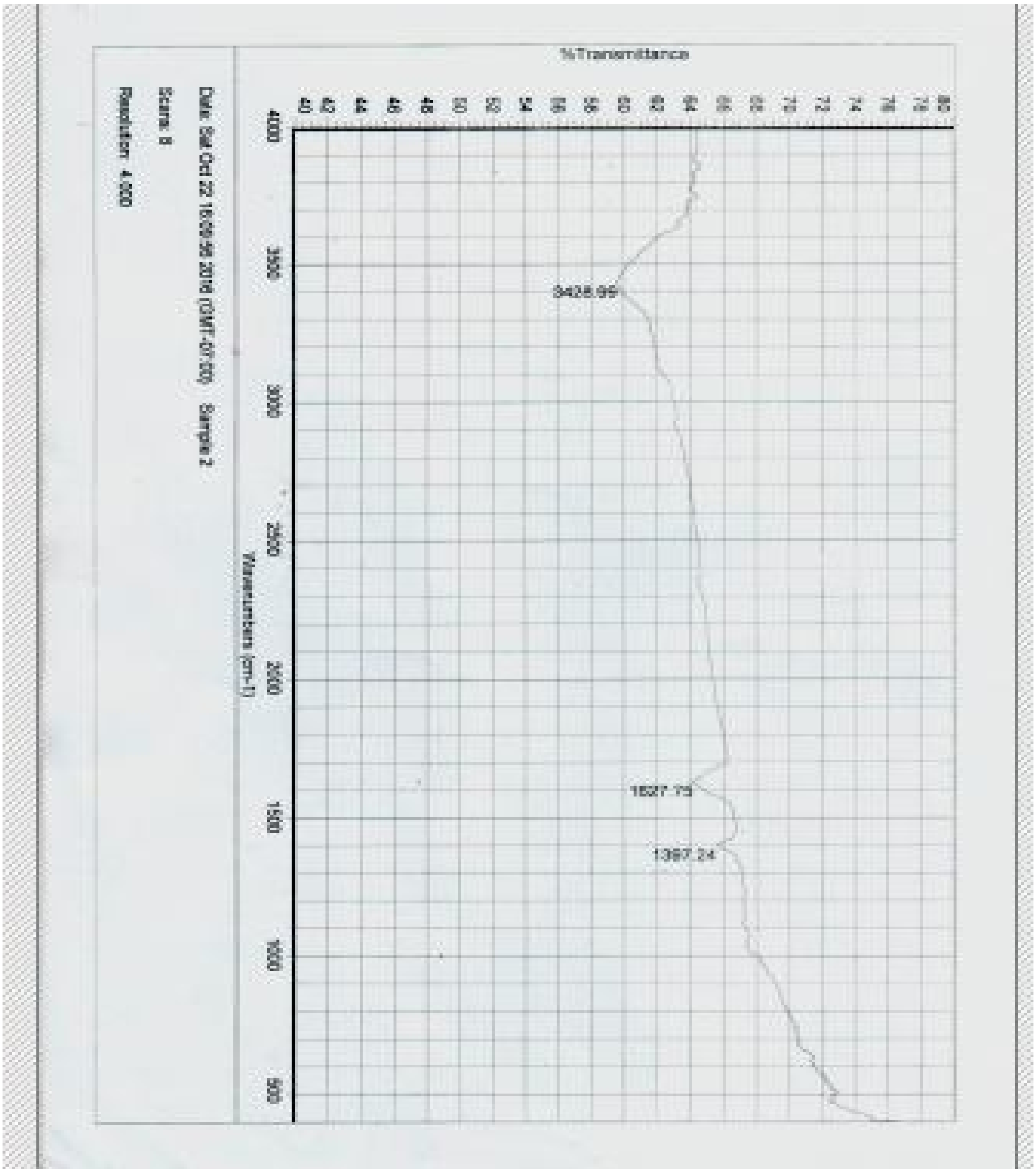


Fig (7):

3.2.2 Infrared spectroscopy of lead:

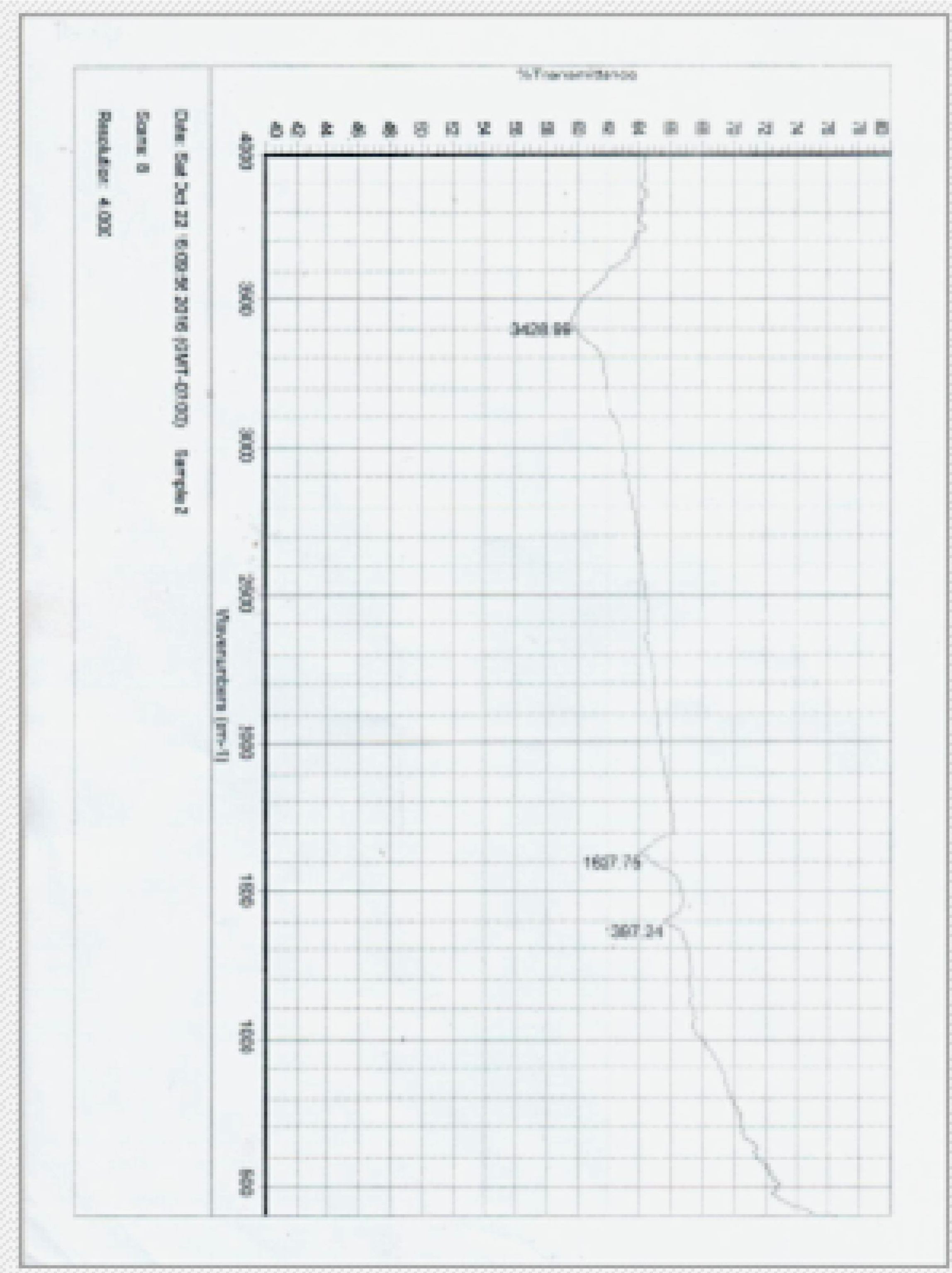


Fig (8)

3.3 Discussion:

In this study, the analysis of samples from five brands by atomic absorption and estimate the concentration of lead in kohl found that the percentage of lead in Kajal is equal 0.3% at 0.0412 ppm , Athmad equal 20.75% at 284.8100 ppm , Nour powder kohl equal 1.1% at 0.1510 ppm , L'Oreal equal 2.5% at 0.3431 ppm , XXL pencil equal 2.1% at 0.2882 ppm . Observing this concentrations of lead in all samples are less than the standard which is 50% .

3.4 Recommendation:

1. Keep the range of concentration of lead in cosmetics less than standard by using few amount of galena.

2. Decreasing of using lead containing cosmetics specially kohl to keep concentration of lead in body or blood stream stable .

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